Gold Plating in the Electronics Industry

A REVIEW OF WORK REPORTED AT THE SUR/FIN 81 CONFERENCE

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Sur/Fin 81 — the Sixty-Eighth Annual Conference of the American Electroplaters' Society (AES) was held in Boston, Massachusetts, from June 28th to July 2nd, 1981. Papers highlighting various aspects of gold electroplating are summarized in this review.

As in previous years, much interest at Sur/Fin 81 was centred on electrodeposition of gold, with cost reduction being a prime factor behind some of the new ideas presented.

Gold Electrodeposition Processes

Two articles dealing with plating processes discussed methods of depositing gold at higher rates than with usual techniques, and in selected areas.

R. Duva (1) reviewed the importance of high-speed selective plating of gold and other precious metals, emphasizing the necessity of designing processes and equipment together. Critical factors when plating gold at high speeds are agitation, to allow adequate metal ion transfer within the cathode film, and anodic oxidation. The author pointed out that for each gram of gold deposited, 0.25 g of cyanide is generated within the electrolyte and disturbing chemical reactions can ensue. He went on to demonstrate the build-up of organic materials in a pure gold electrolyte and the beneficial effects of regular and proper adsorption of this on carbon to ensure deposit quality. An interesting statistic mentioned was that in 1980, at least 70 per cent of the gold coatings electrodeposited in the U.S.A. had been produced under high-speed conditions.

R. J. von Gutfeld (2) presented one of the most innovative topics in many years — the use of laser beams to enhance deposition. His work established that a laser energy of between 10^6 and 10^{10} W/m^2 increases thermal activity in the area of plating, and deposition speeds of 12 μm/s are achieved in localized areas. Selective gold plating of connectors would be a likely application of this effect and certain electroless processes might be carried out selectively using this technique.

Properties of Coatings

A paper discussing the properties and testing of gold deposits was presented by W. R. Bitler of Pennsylvania State University (3). This described the interdiffusion of gold coatings and their substrates. In this AES-sponsored research, the mechanisms involved in the diffusion and the interdiffusion of gold with tin-nickel and cobalt underlayers held at 100 or 175°C for a one-year period were studied. Observations that the diffusion rate is significantly different at these temperatures from that reported at higher temperatures, and that impurities, grain orientation and grain boundary movement all influence this rate were made. The author reported that no diffusion of gold was observed when a barrier was used between the gold and its substrate, that interdiffusion of copper and gold was much less at 100 and 175°C than at temperatures selected for other studies and that no recrystallization of the gold was observed.

The performance of film integrated circuits (FIC's) can be affected by changes in the resistivity of electrodeposited gold layers since these contribute most to the conductivity of titanium/palladium/gold metallizations. Similar considerations apply to other multi-layer systems, such as titanium/palladium/copper/gold and titanium/palladium/copper/nickel/gold. Thus, C. Shiflett (4) investigated the resistivity of gold films applied to a titanium layer on a glass or ceramic substrate, the intermediate layer being necessary to promote adhesion. Pure gold was deposited from a citrate-buffered electrolyte at pH 5.0. The effects of substrate surface roughness, gold film thickness and heating on the resistivity of the films were studied. The ratio of sheet resistance of gold plated over ceramic to that of gold on glass was plotted against thickness. This ratio decreases as thickness increases, reaching an asymptotic value of 1.03 for a gold thickness of approximately 1.5 μm. The resistivity of deposits on glass reaches a minimum value of 2.91 μohmcm for a gold thickness above 1.0 μm. (The resistivity of bulk gold is 2.2 μohmcm.) Heating the deposits lowers the resistivity until a minimum is obtained in 1 000 hours

at 100°C, but in less than 15 minutes at 300°C. The overall conclusions of this work are that increasing surface roughness increases sheet resistance and that this effect is lessened as the gold thickness increases. Rougher substrates also cause higher resistivity since deposits are thinner (for the same mass of gold per unit area) and the electrical path is longer. The resistivity of films is greatest at low thicknesses and is always greater than that of bulk gold. The author considers that this may be a function of grain size or a result of the inclusion of light elements (hydrogen, nitrogen, oxygen, carbon) in the deposits.

The important topic of porosity testing of gold deposits was given consideration in a presentation by C. A. Holden and R. Huik (5). They found that the nitric acid vapour test is suitable for gold deposit thicknesses in excess of 1.9 μm, but gives poor results when thicknesses are less than 1.25 μm. In the latter case, the substrate is severely attacked, which leads to possible misinterpretation of results. An added problem where nickel underlayers are employed is the deliquescent nature of the corrosion product (nickel nitrate). The authors set out to improve and refine this testing technique, so that it could be applied to thin gold deposits and selectively plated parts. The variables studied include acid temperature, age and concentration, time prior to counting pores after testing, degree of bare metal exposure and the method of counting pores. During this work it became obvious that the ambient relative humidity and the size of the desiccator (volume of vapour above the acid) were also critical factors. A post-testing dip in freshly prepared 1 per cent ammonium polysulphide solution was found to overcome the problem of deliquescence with nickel nitrate. This treatment produced well-defined black residues which could be easily counted. Standards were prepared using 0.1 μm of gold over OFHC copper sheet and 0.5 μm of gold over bright nickel. The authors recommend that:

(1) The test should be carried out at less than 60 per cent ambient relative humidity
(2) The nitric acid should be discarded when its strength falls to below 41° Baumé
(3) The ceramic plate of the desiccator should be replaced with a glass fixture to hold the test pieces
(4) A 150 mm internal diameter desiccator should be used
(5) The acid temperature should be 24±4°C
(6) A fluorescent light and appropriate grid pattern should be used when counting pores
(7) Standards should be used to check reproducibility
(8) A sulphide dip should be used after testing when nickel underlayers are present
(9) The test duration should be one hour for gold coating thicknesses of 1.25 μm or greater and 30 minutes for thinner deposits.

Process Control

The use of D.C. plasma echelle grating spectroscopy for the analysis of gold plating solutions and deposits was compared with that of fire assay and atomic absorption spectrophotometry (AAS) in the work described by H. Griffin, A. Savolainen and R. Haschke (6). This relatively new technique offers certain advantages over AAS such as the use of a high temperature plasma (5 000 K) in place of the much cooler AAS flame, resulting in fewer interferences. The sample is prepared in a similar way to that necessary for AAS, but the dilution need not be so great. A pump is used for sample delivery to the plasma rather than the system of gas aspiration employed in AAS instruments. Newly developed polychrometers enable excellent resolution (0.004 nm at 200 nm, and 0.008 nm at 400 nm). Up to 20 metallic elements can be determined very quickly in a single sample and the system can be made almost fully automatic. The cost of this technique was compared to that of fire assay in the paper. The authors' conclusions are that D.C. plasma echelle grating spectroscopy offers similar precision to fire assay, but is more accurate and cheaper to run. The elimination of interferences suffered by AAS and the possibility of automating this new technique offer considerable advantages over traditional methods.

Recovery

The very topical subject of recovery of precious metals from electroplating sources was comprehensively discussed by G. I. Edson (7). All aspects were covered, including the sources, collection, processing and refining of gold and other precious materials. According to the author, the biggest losses occur as a result of poor collection systems. Many users do not collect wipes or filters and, while these are difficult to assay, they may contain considerable amounts of gold. In-house processing was recommended prior to outside refining, but specialized equipment is necessary to obtain optimum results.

References

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2 R. Duva, 'New Developments in High Speed Continuous Plating of Precious Metals', Conference paper
3 R. J. von Gutfeld, 'Laser Enhanced Electroplating and Etching for Maskless Pattern Generation and Circuit Repair', Conference paper
4 C. Shiflett, 'The Electrical Properties of Soft Gold', Conference paper
5 C. A. Holden and R. Huik, 'Critical Nitric Acid Vapor Test Parameters Affecting the Apparent Porosity of Thin and Selectively Plated Gold', Conference paper
6 H. Griffin, A. Savolainen and R. Haschke, 'The Use of D.C. Plasma, Echelle Grating Spectroscopy for the Analysis of Plated Gold', Conference paper
7 G. I. Edson, 'Recovery of Precious Metals for the Electroplating Industry', Conference paper